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News Briefs

General Developments

Inquiries about News Briefs, where no contact person is identified, should be referred to the Managing Editor, Journal of Research, National Institute of Standards and Technology, Building 101, Room E215, Gaithersburg, MD 20899-2500; telephone: (301) 975-3577.

PAST PRECISION MEASUREMENT GRANT HOLDER WINS NOBEL PRIZE

The 2001 Nobel Prize in Physics will be shared by two JILA fellows, Eric Cornell of NIST and Carl Wieman of the University of Colorado, and Wolfgang Ketterle of MIT for their work on Bose-Einstein condensation of atoms.

In 1979, Carl Wieman, then at the University of Michigan, was awarded a NIST Precision Measurement Grant. This brings to three the number of people who as young faculty members received one of our grants and went on to be awarded the Nobel Prize. The two others are Daniel Tsui of Princeton University and Steven Chu of Stanford University. An additional Nobel Prize winner, William Phillips of NIST, is closely related to this group. His Ph.D. work was done on an experiment for which his advisor, Daniel Kleppner of MIT, was awarded a NIST Precision Measurement Grant in 1970.

As part of its research program since 1970, NIST has awarded Precision Measurement Grants to U.S. universities and colleges so that faculty may conduct significant, primarily experimental research in the field of fundamental measurement or the determination off undamental constants. NIST sponsors these grants to encourage basic, measurement-related research and to foster contacts between NIST scientists and U.S. academic institutions actively engaged in such work. The grants also are intended to enable researchers to pursue new, fundamental measurement ideas for which there are few to no other sources of support.

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NIST COMPLETES TWO CIPM KEY COMPARISONS

Two pressure key comparisons, piloted by NIST, under the auspices of the CCM/CIPM, were recently completed. Comparison CCM.P-K4 spanned 1 Pa to 1000 Pa (absolute), and involved seven national metrology institutes (NMIs) using two different principal techniques (liquid-column manometers vs static expansion systems) to realize pressure. Comparison CCM.P-K5 spanned 1 Pa to 1000 Pa (differential), and involved four NMIs using two principal techniques (liquid-column manometers vs double pressure balances) to realize pressure.

These comparisons encompass low to medium vacuum measurements, and are important for accurate metering of low-speed air flows. Besides concluding that the participating NMIs were generally equivalent and that there was no significant relative bias between the principal techniques, these comparisons were notable for several other reasons. Of the six prevailing pressure comparisons in the CCM, none have been completed on-schedule other than these NIST-piloted comparisons.

This was achieved by simultaneously circulating two nominally-identical transfer standard packages, one each for EUROMET and APMP participants, that could be used for both absolute and differential pressure measurements. Transfer standards in this pressure range have historically proven problematic and prone to large shifts. However, the NIST-developed transfer standard packages, based on a combination of high-stability MEMS pressure sensors with high-sensitivity capacitance diaphragm gauges, were robust and reliable, thereby enabling the success of these comparisons. This successful effort has earned NIST recognition for its ability to engineer a first-ever comparison of this magnitude and quality over this challenging pressure range.

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NIST SCIENTISTS DISCOVER AN UNEXPECTED OPTICAL PROPERTY IMPORTANT TO MICROCHIP MANUFACTURERS

Scientists at NIST have determined that calcium fluoride, one of the principal materials used for optics in the ultraviolet spectral region, exhibits an unexpected, intrinsic birefringence in the deep ultraviolet. The presence of the effect has been confirmed both experimentally and theoretically, and its magnitude is sufficiently large that calcium fluoride may cause unacceptably large aberrations in optical components intended for use by chip makers developing advanced 157 nm lithography processes. At a minimum, the industry will need to correct for the birefringence.

Ordinarily, a crystal with a cubic structure such as calcium fluoride does not exhibit birefringence in stress-free material because of its high symmetry. However, when the wavelength of light is only a few hundred times the crystal interatomic spacing, the symmetry-breaking effect of the finite value of the photon wavevector starts to become significant and gives rise to the effect. These findings were reported at two SEMATECH meetings earlier this year. NIST scientists are attempting to nullify the birefringence by using mixed crystals of calcium fluoride and barium fluoride, a substance that exhibits opposite birefringence behavior. For additional information see: http://physics.nist.gov/duvbirefring. [J. H. Burnett, Z. H. Levine, and E. L. Shirley, Intrinsic birefringence in calcium fluoride and barium fluoride, Phys. Rev. B **64**, 241102 (2001).]

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NIST SCIENTISTS IMAGE RING MAGNETS USING SEMPA

Researchers at NIST, in collaboration with the University of Cambridge, Thin Film Magnetism Group, have used the NIST Scanning Electron Microscopy with Polarization Analysis (SEMPA) facility to directly image the magnetic domain structure of mesoscopic ring magnets. These micrometer sized rings and discs, patterned out of cobalt thin-films, are the basis for new types of nonvolatile, magnetic random access memories, and the SEMPA measurements provided the first images of various magnetic structures occurring in the magnets.

Some of the magnetic structures that were observed agree with predictions based on earlier, non-spatially-resolved, magnetization measurements of these films. However, additional, unexpected domain wall structures were also found to exist. Knowledge about the nanoscale magnetic structure of the various magnetic states and how the states switch from one to another provides critical information needed to determine whether these patterned magnetic structures will make useful, reproducible magnetic memories.

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IMPROVED RADIOMETRIC STANDARDS LEAD TO HIGH-ACCURACY MEASUREMENTS

Improvements in detector-based technology over the past decade have opened a new era in the field of metrology for radiometry and photometry. NIST Technical Note TN-1438 was published to summarize the results of the research and development work carried out by NIST on constructing accurate and wide-dynamic-range radiometers with high-performance detectors for use in the ultraviolet, visible, and infrared wavelength regions. Entitled *Optical Radiation Measurement With Selected Detectors and Matched Electronic Circuits Between* 200 nm *and* 20 μm, TN-1438 is a compilation of 11 articles and research papers published by a NIST scientist.

Improved radiometer standards are the key components for several recently realized high-accuracy scales. Using the new radiometers, improved scales have been realized for a variety of important radiometric and photometric measurements, for example, spectral power, irradiance, and radiance responsivity. The improved scales are then transferred to standards used by our customers. The result is lower measurement uncertainty for NIST-traceable optical radiation standards.

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NIST PUBLISHES 2001 EDITION OF SP 330

The 2001 Edition of NIST Special Publication 330, *The International System of Units (SI),* is now available. SP 330 is the U.S. version of the English text of the seventh edition (the most recent) of the definitive international reference on the SI, the modern metric system, published in 1998 by the International Bureau of Weights and Measures (BIPM). The 2001 Edition of SP 330 also incorporates *Supplement 2000: Additions and Corrections to the 7th Edition* published by the BIPM in June 2000.

The main body of NIST SP 330 gives the essentials of the current form of the SI. However, Appendix 1 provides the resolutions, recommendations, and declarations put forward on units of measurement and on the SI since 1889 by the General Conference on Weights and Measures and the International Committee for Weights and Measures. Further, Appendix 2 summarizes the current state of the practical realization of some important SI units. Appendix 3 gives a brief description of the governing bodies established by the Meter Convention, which was signed in Paris on May 20, 1875, by 17 nations including the United States.

Single copies of NIST SP 330 may be obtained from the NIST Metric Program, (301) 975-3690; email: metric_prg@nist.gov. NIST SP 330 is also available online at http://physics.nist.gov/cuu.

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ELECTROMAGNETIC COMPATIBILITY TESTING FACILITY INSTALLED IN FORCE LABORATORY

A NIST scientist successfully led the efforts to procure, prepare, install, and validate an electromagnetic compatibility testing facility for immunity measurements of digital load cells. This facility has been installed as part of the NIST Force Laboratory and consists of an electromagnetically anechoic 7 m enclosure, together with associated instruments for conducting tests for the immunity of digital load cells to radiated electromagnetic fields in accordance with the International Electro-technical Commission as required by International Organization for Legal Metrology R60 specifications for legal metrology testing. Features were incorporated into the system to enable the application of force to transducers while maintaining proper radiated field uniformity.

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NIST AIDS DEVELOPMENT OF NEW BONE GRAFT MATERIALS

Scientists from NIST and research associates from the American Dental Association Health Foundation have demonstrated a new approach to bone regeneration. More than 100 000 bone grafts are performed each year in the United States, but the amount of available autologous bone is limited and available material is difficult to shape. Furthermore, the number of bone grafts will likely increase as the population ages. Thus, the development of a synthetic, moldable bone graft is the primary goal of tissue engineered products industry.

The new approach extends previous work by the American Dental Association Health Foundation at NIST on a self-setting calcium phosphate cement that can be sculpted to fit the contours of a wound. This cement was improved in recent work by making it macroporous and osteoinductive through inclusion of polymer microspheres and a bone growth factor, respectively. The polymer microspheres, called porogens are made of a biodegradable polymer that initially stabilizes the graft, but then can degrade gradually, leaving pores of the appropriate size for colonization by osteoblasts. The calcium phosphate cement matrix would slowly dissolve and be replaced by new bone. The NIST research has shown that in cell media the polymer-modified cement undergoes complete degradation of the porogens, leaving a porous structure. Osteoblast-like cells were shown to adhere, attain a normal morphology, proliferate and remain viable when cultured on the new composite graft. The calcium phosphate cement is further improved by providing controlled release of an osteoinductive protein that facilitates growth of new bone. Studies have shown that adjusting the amount of porogens could modulate the protein release kinetics.

The NIST work has attracted the interest of biotechnology companies that focus on the development of synthetic bone grafts.

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STRESSES IN ELECTRON BEAM WELD JOINTS OF SUPERALLOYS

Single crystalline turbine blades are the pinnacle of a decade-long development and refinement of nickel-based superalloys. Because of the considerable investment in manufacturing, electron beam welding with a polycrystalline replacement material is used to repair minor structural damage of failed components of these otherwise poorly weldable materials. While both the fusion zone and the heat affected zone are small, about one millimeter, the cooling rates are high so that very high residual stresses are generated.

In order to study the efficiency of stress relief, scientists from the NIST Center for Neutron Research and a private company have investigated by means of neutron diffraction the residual stress distribution both in the as-welded state and after a heat treatment. The heat treatment includes a solution treatment and subsequent aging. The typical grain size of the polycrystal side of the weld joint was approximately one millimeter which is about the size of the incident neutron beam. Thus, stress measurements could be done only on the single crystal side of the joint.

It was found that the as-welded sample exhibits tensile stresses on the order of the yield stress both parallel and perpendicular to the weld. We have also found evidence for solution partitioning and a substantial level of plastic strain both in the fusion zone and in an approximately half-millimeter wide heat affected zone. Both effects are completely removed after the heat treatment. The heat treatment also removes very effectively and completely the detrimental tensile residual stresses in the extended weld zone. These results are the first on this type of system, in which single-crystal and polycrystalline constituents are joined.

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PATENT ISSUED FOR REVERSE-ANGLE X-RAY DIFFRACTION

Patent 6,269,144 was recently issued to NIST for a "Method and Apparatus for Diffraction Measurement Using a Scanning X-ray Source." The concept combines the high-energy x-ray diffraction technology that has been developed by NIST during the past 5 years with the unique area-scanned x-ray tube developed by a private company. The technique differs from conventional x-ray scanning systems in that the x-ray source is scanned through a pattern instead of the specimen. The technique is particularly appropriate for rapid determination of texture or identifying the phases present in a specimen.

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INTERNET-ASSISTED, ON-SITE SPECIAL TEST PERFORMED BY NIST

Staff at NIST have completed the first Internet-assisted, on-site special test of an electronic instrument. The test was performed for the Sandia National Laboratories (SNL) and the instrument under test was a multifunction calibrator used to test the five electrical functions available on most digital multimeters (DMMs). The reference at NIST was a similar calibrator, which has been calibrated using basic NIST electrical standard artifacts. This type of artifact calibration is also used at many industrial standards laboratories; however, even with automation, it takes a skilled metrologist about 1 week to perform the tests. The objective of the new service is to relieve NIST customers of this burden by performing an on-site test of their calibrators using a precision DMM, calibrated by a NIST reference calibrator, as a traveling standard. The DMM and the calibrators are fully programmable, and the control software used to calibrate them implements a specific test procedure for each instrument. For the on-site test to be effective, it is critical that the same test procedures are employed at both laboratories. While the concept of this type of remote calibration was proved last year, this is the first instance of an official report being issued.

As a first step, an Internet-based video conference was established between NIST and SNL. SNL metrologists described their test procedures and equipment, including the calibrator under test and the proposed traveling standard. NIST staff described the recommended test procedures and the NIST-developed control software, which had been sent electronically to SNL. The traveling DMM was then tested using both SNL and NIST control software to ensure compatible procedures. After several discrepancies were resolved, the traveling DMM was sent to NIST where it was tested and returned to SNL for follow-up tests.

The uncertainties of the on-site measurement were typically five times better than the calibrator specifications, proving the effectiveness of supporting these instruments in the field. Collaboration with SNL is underway to further improve uncertainties and to simplify the measurement process.

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NIST STAFF DEVELOP MICROWAVE POWER STANDARD FOR SINGAPORE STANDARDS BOARD

NIST microwave power standards consist of microcalorimeters as primary reference standards and bolometric detectors as secondary transfer standards. NIST builds and maintains both standards for its own use and also builds bolometric detectors for other standards laboratories. NIST staff members built a microcalorimeter for the Singapore Productivity and Standards Board (PSB) and delivered it to them in November 2000. It has a Type N coaxial connector and is used for frequencies from 50 MHz to 18 GHz. This connector type is popular among manufacturers and is used in our highest volume calibration services.

The performance of the Singapore microcalorimeter was compared with an earlier microcalorimeter in use at NIST. Two NIST Type N bolometric detectors that were purchased by PSB several years ago were used as transport standards in this comparison. The effective efficiency of the two bolometer mounts was measured in the PSB calorimeter in Singapore in June and in the NIST calorimeter in July. The efficiencies agreed within 0.0015 at all 41 frequencies measured. The expanded uncertainty for the NIST measurement ranges from 0.0024 at low frequencies to 0.0045 at 18 GHz.

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NIST MEASUREMENT IS ADAPTED TO MEET TELECOMMUNICATIONS INDUSTRY NEEDS

To meet the emerging metrology needs of the telecommunications industry, NIST scientists have significantly improved NISTs measurement capabilities for chromatic dispersion. Dispersion causes data pulses in optical fiber systems to broaden, resulting in transmission errors. Previously, NIST produced Standard Reference Material 2524, which was an artifact with a certified zero-dispersion wavelength (ZDW). However, the emergence of wavelength-division multiplexing now requires that chromatic dispersion itself be measured over the ≈80 nm optical bandwidth occupied by multiple wavelength channels. The NIST system has been expanded to make certified measurements of chromatic dispersion with total uncertainties as low as 0.2 %. This improved system also compensates for temperature drift and features a more sophisticated scheme for background removal, cutting the ZDW measurement uncertainty nearly in half. The system can measure a wide variety of the optical fibers found in todays sophisticated networks.

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NIST EVALUATES DYNAMIC SERVICE DISCOVERY PROTOCOLS

Working as part of the NIST Pervasive Computing Program, NIST researchers applied architectural modeling to analyze the quality of service provided by dynamic service discovery protocols under conditions of network failure. Service discovery protocols allow network devices, applications, and services to advertise and discover each other's services to properly complete specific tasks. The protocols currently under investigation include Jini™, Universal Plug and Play, and the Service Location Protocol. NIST shared early results from the work with industry to help improve specifications for commercial products.

To perform their analysis, the NIST researchers used Rapide, an architectural description language developed by Stanford University. They transformed natural-language specifications of specific discovery protocols into architectural models that include network topologies and essential software behaviors. The architectural models are then executed using scenarios devised by the researchers to simulate network degradation and failures. During model execution, the researchers use logical assertions to gauge consistency among states maintained on different nodes. They also use quantitative metrics to assess latencies and overhead associated with restoring the system to a consistent state. The re-searchers produced a paper describing their approach in detail and demonstrating initial results.

The current stage of the project focuses on comparative analysis of generic, alternative architectural models common to different protocols. This work appears to be particularly timely because of the proliferation of service discovery protocols in private industry. Results from this project will help the industry to better understand the logical and performance properties of the current generation of discovery protocols, will help to design improved versions of such protocols in future generations, and will provide practitioners of software architecture modeling with a better understanding of the capabilities and limitations of the research tools available to date.

A website provides a detailed description of the project: http://www.itl.nist.gov/div897/ctg/adl/ sdp_projectpage.html. Another relevant website is http://w3.antd.nist.gov/net_pc.shtml.

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ROOF COLLAPSE STUDIED TO HELP PROTECT FIREFIGHTERS

NIST, in cooperation with the Kinston, NC, fire department, and the Bureau of Alcohol, Tobacco, and Firearms, conducted a house fire test in part to measure the deflection of the roof from ignition to collapse. The feasibility study, funded by the U. S. Fire Administration, examined the potential to measure changes in roof position as an early warning technology for structural failure. Data from the tests are being analyzed and compared with other visual observation of fire conditions to determine if position measurements provide any significant additional information for making decisions about the safety of firefighters who might be on roofs to provide fire ventilation. Venting smoke though roofs is a common practice in fire fighting. It helps clear the smoke from structures to aid other firefighters entering the building. This study follows another conducted in cooperation with the Phoenix fire department to look at the use of thermal imaging as a means to warn about unsafe roof structures.

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NIST ARENAS USED FOR URBAN SEARCH AND RESCUE ROBOT COMPETITION

The NIST reference arenas for evaluating the performance of search and rescue robots were shipped to Seattle, WA, in early August for use in the International Joint Conference on Artificial Intelligence mobile robot rescue competition, which was held jointly with

RoboCup Rescue. The competition is meant to stimulate progress in robot research by providing concrete, repeatable, measurable challenges in sensing, navigation, planning, human-robot interaction, mobility, and other technologies required for successful mobile robots. Robots explore the three different sections that simulate a collapsed building, locate as many victims and hazards as possible, and communicate their findings (preferably with a map) to a human supervisor within 25 min. Victims are represented by mannequin parts, clothing, movement, sound (cries for help, banging), and heat signatures. The arenas provide three different levels of difficulty in navigation, traversability, and spatial layout.

Over the course of 3 days, four teams competed officially and half a dozen different organizations gave demonstrations of their robots going through the arenas. None of the teams accumulated enough points to place, but two qualitative awards were given out. The Artificial Intelligence (AI) Award was given to Swarthmore College for demonstrating the most AI functionality. The platform award was given to Sharif University of Iran for their original tracked vehicle.

The main designer of the arenas, a NIST engineer, was asked to chair the RoboCup Rescue event next year, which will be held in June in Japan. The RoboCup organizers plan to recreate the NIST arenas at each country where the competition is held and leave them behind permanently to stimulate progress. RoboCup Rescue is a new event within the overall RoboCup competition framework. RoboCup is an international effort to foster artificial intelligence and robotics research by providing a standard problem where a wide range of technologies can be examined and integrated. The first problem focus area has been robotic soccer. Wildly popular world-wide, there are more than 3000 researchers from 35 countries participating in robotic soccer matches currently. Due to the Kobe earthquake, Oklahoma City bombing, and other disasters, the RoboCup community selected the search and rescue mission as their second application domain, one with significant potential societal benefits.

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PROPERTIES OF IONIC LIQUIDS—NEW SOLVENTS FOR GREEN CHEMISTRY

Ionic liquids are a class of organic salts that are liquid at or near room temperature. Because they are non-volatile and non-flammable, they have been proposed as recyclable "green solvents." The physical properties of ionic liquids may be tuned, for example, by altering the hydrophobicity, to affect reaction rates and selectivities. However, in spite of the many advantages that these fluids are predicted to offer, fundamental data on their physical and chemical properties are scarce. To provide U.S. industry with the knowledge base to exploit these solvents, NIST researchers have prepared several ionic liquids and have begun to study their effects on rate constants of fundamental chemical reactions.

Rate constants for several oxidation and reduction reactions were measured by the pulse radiolysis technique and compared with the rate constants for the same reactions in other solvents. The rate constants in several ionic liquids are much lower than those determined in aqueous solutions and close to those measured in low-polarity organic solvents such as ethanol. These low rate constants suggest a high degree of ion-association in the ionic liquids. On the other hand, the activation energy of the reaction is close to that measured in aqueous solutions but higher than that in alcohols. These two apparently conflicting results are explained by considering the microenvironments of the reactants, i.e., that each molecule and each site of a large molecule may be solvated by different ions of the ionic liquid.

Rate constants for several reduction reactions also are lower in ionic liquids than in water or alcohols. A striking difference (by four orders of magnitude) was found in one case, due to a combination of the effect of the ionic liquid on the rate of reaction and its effect on the reduction potentials of the two reactants, which in turn affect the rate constants. The effect of ionic liquids on reduction potentials is currently being assessed.

Results of this work can be found in the following references: J. Phys. Chem. A **105**, 7607-7614 (2001) and J. Phys. Chem. A **106** (April 2002)..

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SHAPE-SENSITIVE LINEWIDTH MEASUREMENT PASSES ITS FIRST TEST

With support from International SEMATECH, NIST researchers have been developing a method for obtaining edge shape as well as line width information from images of silicon features (e.g., lines, transistor gates) in integrated circuits. The problem addressed by the new method is particularly acute when nanometer scale dimensional uncertainties are required: a scanning electron microscopy (SEM) image is a distorted representation of the feature. It is a two-dimensional intensity pattern derived from the interaction of an electron beam with a three-dimensional object. How does one deduce the shape and size of the object giving rise to the observed image?

The method works by comparing the measured image of a feature to a library of calculated images for similar features, each differing slightly in size or shape. The images in the library correspond to a range of possibilities and may be interpolated for better resolution. The closest match is deemed to be the shape of the feature that produced the measured image. This is a plausible scheme, but does it really work?

To find out, a comparison was performed this month between the shape of a line determined from a top-down SEM image using the above method with an SEM image of the same line after cross sectioning. In this first test of the shape-sensitive linewidth measurement system, the two measurements agreed to within better than 1 nm on the line's width (174.5 nm). This is probably fortuitously close, inasmuch as both measurements have uncertainties of a few nanometers. They agreed to within a few tenths of a degree on the sidewall angles. Similar agreement was achieved in a comparison on a second line. Still under investigation, there was disagreement about the roundness of the upper corners, an aspect of feature shape that shows up in the image in only a subtle way.

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NIST DEVELOPS AN IMPROVED MODULAR RADIOMETER/PHOTOMETER SYSTEM

A new generation photo/radiometer system has been established to realize and propagate the high accuracy detector-based radiometric scales maintained by NIST. The radiometers can measure a variety of radiometric quantities such as radiant power, irradiance, and radiance over spectral regions from the ultraviolet to the near infrared. The photometers measure illuminance and luminance.

The new radiometers and photometers are modular systems. The detectors can be cooled to improve the noise performance and the stability of the meters, while the filter packages, positioned in front of the detectors, can be heated slightly above room temperature to ensure stable spectral transmission without condensation. A variety of photodiode detectors (e.g., nitrided-Si, Si, Ge, InGaAs, and extended InGaAs) are positioned behind the different filter and diffuser combinations to cover the spectral regions over which the instrument is sensitive. Typical relative standard uncertainties of radiometric and photometric measurements made using the new standards are 0.1 %.

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MOBY: AFLOAT IN HAWAII

A Marine Optical Buoy (MOBY) off the coast of Lanai in the Hawaiian Islands is the centerpiece of the primary ocean measurement site for calibrating satellite-born ocean color sensors. The project, which is being led by NOAA with the support of NASA as well, includes team members from the Moss Landing Marine Laboratory, the University of Miami, San Diego State University, and NIST. Measurements from this ground-truth site are being used to calibrate satellites operated by the United States, Japan, France, and Germany that are designed to infer what is going on at the ocean surface.

The ocean buoy has specialized, optical radiation instruments that measure both the incident solar flux and the water-leaving radiance at sea level and at depths of 1 m, 5 m, 9 m, and 12 m. The instrumentation features two imaging spectrometers, onboard calibration sources, and eight light collectors multiplexed with fiber optical cables.

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NIST RESULTS LEAD TO IMPROVEMENT IN ZENER VOLTAGE REFERENCE

Measurements made at NIST for the characterization of Zener voltage standards have enabled the manufacturer to redesign their instruments for improved behavior. As part of NIST's efforts to develop a 10 V measurement assurance program (MAP), NIST undertook an extensive study of the characteristics of eight Zener voltage standards under a wide range of differing environmental conditions. One striking feature of the measurements was that all units showed a strong correlation between the Zener voltage output and relative humidity. The time constant for response to humidity change was relatively short, on the order of about 2 to 3 weeks. This behavior would have seriously limited the use of these instruments as transport standards for the MAP.

The NIST measurement results were shared with the manufacturer in hope that the detailed characterization might point to the source of sensitivity to environmental humidity. A preliminary analysis identified certain epoxy-encapsulated resistors as likely candidates for the problem. When relative humidity changed, the plastic molding would either absorb or desorb water. The resulting swelling or shrinking would change stress patterns across the resistor array chip. Numerical simulation predicted resulting voltage changes in reasonable agreement with the NIST measurements. A retrofit to the product line was designed and imple-

mented on four of the NIST units as a test. Subsequent measurements at NIST confirmed a large reduction in the sensitivity to humidity changes, greatly improving the potential for use of these devices as transfer standards for the NIST 10V MAP. The combination of critical measurement data from NIST and the manufacturer's timely response resulted a significant improvement in the performance of these high precision voltage standards.

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NIST RESEARCHERS CHARACTERIZE THE TEMPERATURE-DEPENDENT DIELECTRIC PROPERTIES OF POLYMER MATERIALS TO AID THE ELECTRONICS INDUSTRY

NIST staff members have completed dielectric measurements on a selection of plastic materials. The goal of the research was to obtain well-characterized, variable temperature measurements on a wide array of polymer materials and to develop new characterization metrology. Low-loss plastics are widely used in the microelectronics industry; temperature-dependent measurements are crucial for accurate modeling and design. For example, printed wiring boards generate heat as the computer dissipates energy, so it is very important to know the temperature-dependence of the permittivity of the wiring board.

The features that make these measurements unique include the wide array of materials, the broad temperature range, the use of very precise temperature-dependent thermal data. These measurements required a detailed uncertainty analysis which was lacking in the literature. The materials in the study were selected on the basis of utility in microelectronics applications. These plastics included commercially available materials such as Teflon, Rexolite, nylon, FEP, polyethylene, polysulfone, and others. The temperatures spanned were from –150 °C to 150 °C, the measurement frequency was 10 GHz, and the measurement fixture was a TE01 dielectric resonator. Literature values for the temperature-dependent thermal expansion coefficients were used in the study. The environmental chamber used to achieve the temperature dependence has bulkhead adapters on the sides for insertion of coaxial feeds for the cavity. The chamber was purged with nitrogen gas to reduce oxidation and water vapor.

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PULSED INDUCTIVE MICROWAVE MAGNETOMETER DEVELOPED AT NIST

As part of its program in high-speed magnetics, NIST has developed an automated, pulsed inductive microwave magnetometer (PIMM) to characterize magnetic thin films. The PIMM is designed to measure the magnetodynamical properties of materials used in recording heads for magnetic data storage. The data storage industry is developing new magnetic alloys with high saturation magnetization to use in write heads. The magnetic damping behavior of these new alloys will determine their usefulness for high-speed recording.

The PIMM uses a co-planar waveguide as both a source of fast, pulsed magnetic fields and as an inductive flux sensor. Magnetic field pulses are provided by a 10 V, 55 ps rise-time pulse generator. Orthogonal Helmholtz coils provide the magnetic bias and saturating fields required for the measurement. A 20 GHz digital sampling oscilloscope is used to acquire the data. The system can measure dynamical behavior as a function of several variables, including applied bias field, pulsed field amplitude and width, and sample orientation. Using fast Fourier transforms, the PIMM can determine the frequency dependence of the complex magnetic permeability as well as the step and impulse responses of magnetic systems.

The PIMM includes components necessary for completely automated magnetodynamic measurements. No user intervention is required to insert or remove attenuators on the front end of the high-bandwidth sampling oscilloscope. This reduces the chance of an electrostatic discharge that could damage the sensitive front-end circuitry of the oscilloscope. It also increases the system sensitivity since the attenuators can be set in finer increments to give the largest signal as the pulse amplitude is adjusted.

This unique magnetometer has been used by visiting scientists from university data storage research centers and disk drive manufacturers.

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SMOKE DETECTOR TESTS IN KINSTON, NC

During September 2001, a NIST team of engineers and technicians instrumented and burned a two-story, single-family home in Kinston, N.C., in order to characterize the performance of different types of smoke detectors. Smoke detector arrays were located in the hallway and bedroom upstairs as well as exit paths

downstairs. Each smoke detector array consisted of a number of detectors including photoelectric, ionization, photo/ion combination, aspirated, carbon monoxide, and mechanical heat sensor. Additional instrumentation included smoke meters, thermocouple arrays, and carbon monoxide, carbon dioxide, and oxygen analyzers.

Each fuel package, either a mattress in the upstairs bedroom, an upholstered chair in the living room, or a frying pan of oil in the kitchen, was ignited and the movement of the smoke was monitored as it moved throughout the house. The overall purpose of this project is to determine if different types of fire alarms can respond to threatening residential fire settings in order to permit egress of typical residential structures.

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FIRE SUPPRESSANT DISPERSION IN A COLD AIRCRAFT ENGINE NACELLE

Halon 1301 is the predominant fire suppressant used to extinguish aircraft engine-nacelle fires. Due to its adverse effect on the ozone layer, halon 1301 has been banned from production in the United States since 1994 in compliance with the Montreal Protocol on Substances that Deplete the Ozone Layer. NIST has since been extensively involved in the search for the replacements for halon 1301. Trifluoroiodomethane (CF3I) has been proposed as a potential candidate for halon 1301 in aircraft engine nacelles.

One current focus at NIST is on the dispersion and distribution of CF3I at temperatures below its normal boiling point (–22 °C). Under such conditions, which are encountered during cold start of an aircraft engine on a cold tarmac or high-altitude cruising, there is a potential deterioration in the dispersion of the suppressant and its transport to the fire zone.

An engine nacelle simulator was built to conduct the research. The simulator, with observation windows and measurement ports, has a configuration and dimensions commensurate with a typical small engine nacelle. To simulate low temperature conditions, the entire facility was housed inside a large environmental test chamber in the U.S. Army CECOM Research, Development and Engineering Center at Fort Belvoir, and the suppressant discharge experiments were performed inside the chamber. The dispersion effectiveness of CF3I was assessed based on concentration measurements inside the engine-nacelle simulator using fast-response fiber-optic-based UV spectormeters. Discharge experiments at room temperature were also conducted to establish baselines for comparison.

The concentration measurements indicated that significant reduction in the dispersion effectiveness of CF3I occurred when it was used at temperatures below its normal boiling. Then a fire suppression system designed based on room-temperature test data may fail to provide adequate fire protection. Other parameters that could affect the dispersion and transport of the fire suppressant are currently being examined in our laboratory. Means to improve the performance of the fire suppressant in low temperature applications are also being explored. Extension to other high boiling-point liquid fire suppressants is being planned.

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IMPROVED TECHNIQUE FOR CHARACTERIZATION OF TEXTURE IN THICK PLATES

Commercial metal alloys used for structures are usually produced by rolling or extrusion into the form of plates, bars, or beams. These fabrication processes leave their mark on the material in the form of a texture or preferred orientation of the grains in the microstructure. In many applications, such as the nondestructive inspection and materials characterization of structural components, a knowledge of this texture is of vital importance.

It can be measured in the laboratory by x-ray techniques or by ultrasonic methods that involve precision machining of a specimen cut from the part. However, the information on texture is often needed in the field, at depths below the penetration of x rays or on parts that cannot be cut up. For several years, NIST has been developing ultrasonic measurement procedures for deducing the texture of materials using specimens carefully machined from the part. We have developed a texture measurement technique by combining electromagnetic acoustic transducers (EMATs), which excite and detect ultrasonic waves that propagate near or on the surface of a plate, with theoretical models for the elasticity of polycrystalline aggregates.

It is nondestructive and applicable to large objects found in the field, at rolling mills, or in foundries. It is being used to measure surface residual stresses that distort parts being machined from thick plates, to characterize ultrasonic reference blocks used to calibrate ultrasonic inspection systems, and to monitor the condition of surface layers used to extend fatigue life or improve wear resistance.

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NEW TEST CAPABILITY PROBES FAILURE PROCESSES IN POLYMER COMPOSITES

A new measurement tool for visualizing and quantifying time-dependent failure mechanisms in polymer composites has been developed at NIST. The new test method produces optical images of multi-fiber test specimens as the specimens are deformed under mechanical load. Previous work, based on single-fiber tests, ignored how rupture of one fiber affects the failure of neighboring fibers and provided only limited information regarding the influence of the polymer matrix on mechanical failure.

Initial results from the new test apparatus show that the nucleation of critical flaws in unidirectional fibrous composites depends on the time-dependent redistribution of the mechanical stress in the polymer matrix. The behavior of the matrix determines how the rupture of one reinforcement fiber affects the integrity of neighboring fibers. In addition, new information regarding the role of matrix cracks and residual curing stresses on fibrous composite failure behavior has been revealed. A visual representation of the time dependent failure behavior in two-dimensional multi-fiber composites can be found at: http://polymers.msel.nist.gov/researcharea/multiphase/ project-detail.cfm?PID=56

Knowledge of the time-dependent failure processes in fiber-reinforced composites is critical to their use as structural parts in automotive applications. Automotive design engineers need better tools for predicting and managing how composite parts respond to crashes. To address this need NIST researchers are adapting the object oriented finite element analysis (OOF) program to include viscoelastic materials. The data from the multi-fiber tests will be used to aid the development of failure models for the new OOF program.

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DOMAIN WALLS IN AF/FM MAGNETIC BILAYERS

The exchange coupling between a ferromagnet (FM) and an antiferromagnet (AF) creates a magnetic bias field on the ferromagnet and thereby controls its magnetization characteristics. Discovered more than 40 years ago, this perplexing phenomenon has been intensely studied in recent years, and has been incorporated in the new high sensitivity computer disk read heads that have enabled multiple-gigabyte hard disks.

Using the NIST-developed magneto-optical indicator film technique, NIST researchers have observed directly for the first time the antiferromagnet domain walls and the evolution of a special type of hybrid domain wall in exchange-coupled FM/AF bilayers. We accomplished this using special samples demagnetized at high temperature and cooled to room temperature in zero field. In such samples, we discovered the presence of a hybrid domain wall consisting of coincident ferromagnet and antiferromagnet sections. Under an applied magnetic field, the ferromagnet domain wall moves while the antiferromagnet wall remains stationary. In the process, an exchange spring develops that connects the moving ferromagnetic and the stationary antiferromagnetic domain walls.

As a consequence of the winding and unwinding of the exchange spring during the backward and forward magnetization reversals, a shifted hysteresis loop is observed. These results should enable magnetic recording disc manufacturers to prepare more reliable and controlled devices since now they know what magnetic features to control and how to examine them.

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NIST CHERRY ROAD CD INCORPORATED INTO COMMAND SCHOOL CURRICULUM

Command School is a 3 day training symposium sponsored by the International Association of Fire Chiefs for fire department officers. The objective of the course is to improve fire incident command strategies to reduce firefighter deaths. The Command School instructors developed a presentation utilizing the NIST produced CD-ROM, *Simulation of the Dynamics of the fire at 3146 Cherry Road NE, Washington, DC, May 30, 1999,* NISTIR 6510. Two NIST models, the Fire Dynamics Simulator and Smokeview, were used to simulate a townhouse fire that claimed the lives of two Washington D.C. firefighters. Approximately 2000 fire officers attend Command School each year. Through support from the U.S. Fire Administration, each student will receive a copy of NISTIR 6510 on CD-ROM.

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NIST RELEASES NEW RESOURCE FOR COMPUTER FORENSICS COMMUNITY

NIST released Version 1.0 of the National Software Reference Library (NSRL) Reference Data Set (RDS) to the computer forensics community on Oct. 30, 2001. Version 1.1 is due to be released by mid-March 2002. The RDS is a resource for law enforcement, corporate, and other organizations investigating crimes involving computers. It contains over one million file profiles from software including operating systems, database management systems, word processing packages,

graphics applications, utilities, image libraries, font libraries, and many other types of software. These profiles can be used to "fingerprint" known files on computer systems and either remove or include them automatically in reviewing files for probative content.

The NSRL is supported by the U.S. Department of Justice's National Institute of Justice, the Federal Bureau of Investigation, the Defense Computer Forensics Laboratory, the U.S. Customs Service, the Technical Support Working Group, and NIST.

The RDS is available on CD-ROM from NIST's Standard Reference Data Office. Annual subscription ordering information can be found at http://www.nist.gov/srd/nistsd28.htm.

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NOVEL COMPUTATION ENABLES BEST-YET ESTIMATE OF GROUND STATE OF NEUTRAL HELIUM

A NIST scientist, in collaboration with Indiana University, has computed the nonrelativistic energy for the ground ^1S state of neutral helium to be –2.9037 2437 7034 1195 9829 99 a.u. This represents the highest accuracy computation of this quantity to date. Comparisons with other calculations and an energy extrapolation yield an estimated uncertainty of 10^{-20}.

Exact analytical solutions to the Schrödinger equation, which determines such quantities, are known only for atomic hydrogen and other equivalent two-body systems. Thus, solutions must be determined numerically. How this best calculation to date was accomplished is in an article submitted to the *International Journal of Quantum Chemistry*. To obtain this result, very large basis sets must be used. In this case, variational expansions of the wave function with 4648 terms were employed, leading to the need for very large computations. Such large expansions also lead to problems of linear dependence, which can only be remedied by using higher precision arithmetic than is provided by standard computer hardware. For this computation, 192 bit precision (roughly 48 decimal places) was necessary, and special coding was required to simulate hardware with this precision.

Parallel processing was also employed to speed the computation, as well as to provide access to enough memory to accommodate larger expansions. NIST's Scientific Computer Facility cluster of 16 PCs running Windows NT was utilized for parallel computation. Typical run times for a calculation of this size about are 8 hours on a single CPU, but only 30 min to 40 min on the parallel processing cluster.

This work employs a very novel wave function, namely, one consisting of at most a single r_{12} raised to the first power combined with a conventional non-orthogonal configuration interaction basis. The researchers believe that this technique can be extended to multielectron systems. Work is in progress, for example, to see what uncertainties can be obtained for atomic lithium, which is estimated to require a 6000-fold increase in CPU requirements to reach the same level of precision, making the use of parallel programming techniques even more critical.

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NIST PARTNERS WITH NSA TO VALIDATE SMART CARD SECURITY REQUIREMENTS

Smart cards—credit card-sized plastic cards with a small embedded computer chip that can process or store information—just got smarter and safer, thanks to a NIST collaboration with the National Security Agency (NSA), in which the two agencies formally recognized a major advance in the security of smart card technology. Through their joint National Information Assurance Partnership (NIAP), the agencies issued an evaluation certificate on a formal set of smart card security requirements. These specifications will allow manufacturers to have their smart cards tested to ensure they meet certain security standards. Smart cards have to be protected against hackers because they contain computerized information.

NIST and NSA worked with an international consortium of leading financial services companies to develop security standards for smart cards. The consortium, known as the Smart Card Security Users Group (SCSUG), developed the security specifications using the new international security standard ISO/IEC 15408, which is known as the Common Criteria. A commercial testing laboratory evaluated the specifications, which then were validated by NIST and NSA under the NIAP Common Criteria Evaluation and Validation Scheme. These results will be recognized by the 13 other nations who are signatories of the International Common Criteria Recognition Agreement.

The development and evaluation of SCSUG smart card security requirements represents a successful industry-government partnership contributing to the security of information systems and networks in the United States and around the world. For more information on the Common Criteria Project, including the completed smart card evaluation and validation, see http://csrc.nist.gov/cc/sc/sclist.htm.

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NEUTRON METHODS LINK MICROSTRUCTURE TO PROCESSING AND PERFORMANCE FOR THERMAL BARRIER COATINGS

Although coatings are used in the electric utility and aircraft industries to protect advanced gas turbines from increasingly high operating temperatures, there is presently no single industrial technique that can quantify the component void microstructures that control thermal barrier coating performance and reliability. NIST researchers have developed advanced small-angle neutron and x-ray scattering methods which, with appropriate models, provide a microstructure characterization for thick, free-standing material. Two innovations now extend these studies to submillimeter-thick coatings *in situ* on the substrate, and suggest that such characterization can provide calibration and validation of the partial information available from other methods.

In near-surface small-angle neutron scattering (NS-SANS), reflection geometry is used to determine apparent internal void surface area distributions within thermal barrier coatings to typical mean depths of 0.1 mm. The method has been used to study the effects of thermal cycling on an yttria-stabilized zirconia plasma sprayed thermal barrier coatings deposited on a nickel superalloy substrate. The NS-SANS measurement has revealed a marked increase in intra-splat cracking perpendicular to the substrate, arising from thermal mismatch strains between the coating and the substrate. This effect contrasts with a preferential sintering of intra-splat cracks for free-standing deposits and it significantly modifies the properties and performance of thermal barrier coatings subjected to elevated service temperatures.

For 0.3 mm to 0.4 mm thick thermal barrier coatings on substrates, microstructures can also be determined using a modified form of ultra-small-angle x-ray scattering (USAXS), suitable for the study of anisotropic materials. It has been applied to thermal barrier coatings fabricated by plasma spray and by electron-beam physical vapor deposition.

By combining conventional SANS studies of free-standing thermal barrier coating microstructures with NS-SANS and anisotropic USAXS studies of the modifications introduced when the thermal barrier coating is *in situ* on the substrate, a valuable validation tool is becoming available to support industrial thermal barrier coating design.

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"PHASE FIELD" MODEL OF ELECTRODEPOSITION

NIST metallurgists have for the first time applied the phase field method to the modeling of electrochemical processes. This method employs a phase-field variable, a function of position and time which describes whether the material at a certain location is a particular phase, such as liquid or solid. The behavior of this variable is coupled to the relevant transport equations for the material during electrodeposition of metals, such as copper for the wiring in integrated circuits. Interfaces between phases are described by smooth, but highly localized, changes of this variable. This approach avoids the mathematically difficult problem of applying boundary conditions at an interface whose location is part of the unknown solution. The phase field technique has been developed and applied with great success over the last decade, both by NIST researchers and others around the world. The range of problems addressed includes the time evolution of complex solidification morphologies related to the casting of metals, the behavior of crystalline dislocations under stress, and surface electromigration on metals.

This new model also predicts the behavior of electrical charges at the electrode-electrolyte interface. The resulting relationships between electrostatic potential and surface energy (electrocapillary curves), surface charge, and differential capacitance are completely consistent with the traditional sharp-interface models of electrochemical interfaces. This new phase field method provides advantages over existing sharp-interface models in that details of interfacial behavior can be readily explored on complex morphologies, such as within the narrow trenches used in microelectronic fabrication or the dendrites formed during battery recharging.

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A MODEL SYSTEM FOR SCATTERING STUDIES OF MEMBRANE PROTEINS

Because of the difficulty in crystallizing membrane proteins, there is considerable interest in identifying systems that mimic biological membranes and facilitate structural studies of inserted proteins. Solutions of lipid-rich mixtures of phospholipids with certain detergents have shown promise in this regard. Over a range of conditions, these mixtures form isolated bilayer fragments that are stabilized by the segregation of the

detergent molecules at the edges. The interior of these disk-shaped single-bilayer structures thus provides an environment for guest proteins that is topologically and chemically similar to that of cell membranes. It has also been possible to insert chelated magnetic ions into the bilayer fragments to align the bilayer normals parallel to an applied field, which in turn can be used to orient the fragments, and any inserted proteins, for a scattering experiment.

To assess the potential of using magnetically-doped, mixed lipid bilayers for structural studies of membrane proteins, we have carried out extensive small-angle neutron scattering (SANS) measurements of the morphology, phase behavior, and magnetic alignment of phospholipid detergent mixtures. These data have revealed a previously unrecognized phase at temperatures above 40 °C in which the bilayer fragments coalesce to form extended single-bilayer sheets. In this phase, the sheets readily align in moderate fields. Measurements in the aligned state reveal that the bilayer sheets are not homogeneous but have defects, probably due to perforations lined with the short chain detergent molecules. By reducing the detergent fraction by about 30 %, we have extended the region of stability of this phase to physiologic temperatures while maintaining its basic structure and degree of alignment.

Protocols for inserting membrane proteins in this, the most promising membrane support system identified so far, are currently being developed to enable SANS measurements of the conformation the proteins have in actual cell membranes.

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BREAKTHROUGH IN SUB-10 nm NANOFABRICATION IN SILICON

NIST researchers in the atom-based dimensional metrology project made a significant breakthrough in the processing and fabrication of atomically flat and ordered silicon surfaces. The researchers have written features of critical dimensions as small as 10 nm in silicon. This process has now been repeated several times and can be considered a controlled process. This work is focused on developing the means to perform nanometer-scale surface modifications reproducibly and to develop new metrology methods for calibration and characterization on the nanometer scale. These recent developments involved the controlled desorption and breaking of hydrogen surface bonds to create stable structures where complex forms and even words can be written in a space of only 100 nm.

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SUCCESSFUL POLISHING PROCESS MAKES PROGRESS TOWARD MEMS CAPABILITY

A NIST scientist succeeded in using a chemical mechanical polishing methodology developed at Stockton College, NJ, to polish tungsten-coated silicon wafers. The specific need is for a high quality, uniform optical polish as a means of establishing the features of a silicon-based micro-electrical-mechanical system (MEMS). This advancement in the polishing process is significant in that it is one of several critical and challenging steps in a multi-laboratory effort to develop and demonstrate an internal NIST capability to produce MEMS devices.

The initial NIST test case and objective is a MEMS hotplate. During a 1999 sabbatical at NIST, a Stockton College staff member developed the polishing approach and process parameters as a means of precisely controlling the weight of prototypes and tungsten-based mass standards. The NIST scientist made the necessary connection between the earlier work and the current need for polishing silicon-based artifacts. Notably, prior to these results, the plan was to use a commercial process for this task that was proving very difficult.

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PRECESSIONAL SWITCHING IN MAGNETIC MEMORY DEVICES DEMONSTRATED BY NIST

A particular type of thin-film magnetic device called a spin-valve can be engineered to have two stable states of electrical resistance based on the relative magnetization orientation of its ferromagnetic layers. This property has motivated a strong interest in using spin-valves as recording bits in non-volatile, magnetic random access memory (MRAM).

A primary technical hurdle is precise control of the switching of individual devices. NIST researchers have been studying the dynamics of magnetization reversal in spin-valves. Devices have sub-micrometer dimensions

and are fabricated within a test structure that includes high-bandwidth transmission lines. One line delivers ultra-fast magnetic field pulses to the device. The other line is electrically connected to the device and carries the voltage pulse generated as the device changes state. This voltage pulse serves as a probe of the magnetization dynamics of the device.

In a spin-valve, only one ferromagnetic layer, the free layer, responds to external fields. Internal magnetic fields within the device allow only two stable magnetization directions, 180°, along an easy axis. Current implementation of MRAM requires field pulses applied for 10 ns to 20 ns along either the positive or negative easy axis, depending on the desired state. The NIST scientists have discovered a way to switch the devices using field pulses of less than 300 ps duration directed perpendicular to the easy axis. The magnetization is reversed due to large-angle precessional motion. For longer duration pulses, the device does not switch because the magnetization rotates back to its initial direction while the pulse is on.

Precessional switching requires only a single polarity pulse applied perpendicular to the device easy axis, which results in a toggle operation of the magnetic state of the device. This is a simpler and more efficient bit-setting operation than using pulsed fields along the easy axis which requires longer pulses in both directions.
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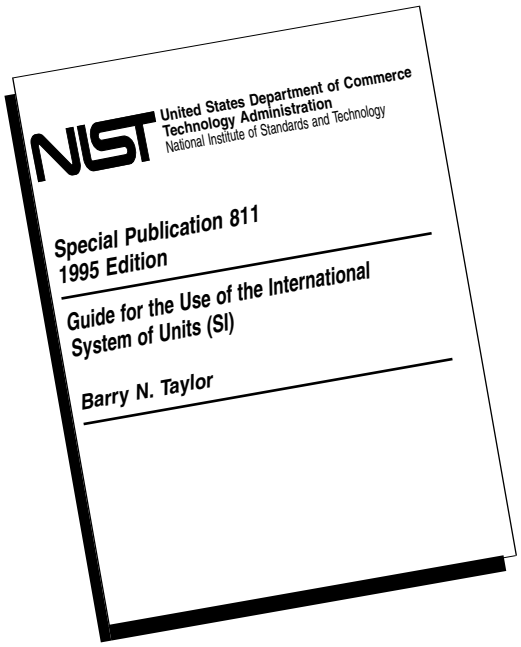
The International System of Units (SI)

A Guide for the Use of the Modern Metric System

NIST Special Publication 811, 1995 Edition



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The 1995 Edition of the National Institute of Standards and Technology Special Publication 811, *Guide for the Use of the International System of Units (SI)*, by Barry N. Taylor, is now available.

The 1995 Edition of SP 811 corrects a number of misprints in the 1991 Edition, incorporates a significant amount of additional material intended to answer frequently asked questions concerning the SI and SI usage, and updates the bibliography. The added material includes a check list for reviewing the consistency of written documents with the SI. Some changes in format have also been made in an attempt to improve the ease of use of SP 811.

The topics covered by SP 811 include:

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Single copies of the 84-page NIST SP 811, 1995 Edition, may be obtained by contacting the NIST Metric Program, 100 Bureau Drive, Stop 2000, Gaithersburg, MD 20899-2000; telephone: 301-975-3690; fax: 301-948-1416; email: metric_prg@nist.gov. NIST SP 811 is also available online at the NIST Web site entitled “NIST Reference on Constants, Units, and Uncertainty,” physics.nist.gov/cuu.

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